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Title of Invention	FLUORINATED SULFONAMIDES AS LOW-FLAMMABILITY SOLVENTS FOR USE IN ELECTROCHEMICAL CELLS



Abstract

The invention relates to the used fluorinated sulfonamide as the flammability solvent lowering in the electrochemical cell electrolyte.



Representative Drawing(s)

Fig. 1



Description

■ Brief Explanation of the Drawing(s)

Figure 1 is profile showing the experimental result about the electrochemical stability of the N,N-dimethyltrifluoromethylsulfonamide according to the working example 6 of the present application.

■ Details of the Invention

■ Purpose of the Invention

■ The Technical Field to which the Invention belongs and the Prior Art in that Field

The invention relates to the fluorinated sulfonamide as the flammability solvent lowering the electrolyte for for using in the electrochemical cell.

The lithium ion battery (batteries) has among the system which most usefuls as the car use. The use field was expanded to the battery of the vehicles for electrically driven from electronic product (example, the mobile phone, and camcorder) of the chronic disease.

This battery is composed of the cathode, anode, and separator and nonaqueous electrolyte. The used cathode, typically, $\text{Li}(\text{MnMe}_2)\text{O}_4$, $\text{Li}(\text{CoMe}_2)\text{O}_2$, $\text{Li}(\text{CoNiMe}_2)\text{O}_2$ In other words, the other lithium-intercalation compound. Anode is composed of the lithium metal, carbon material, graphite, the graphitic carbon material, the other lithium-intercalation compound, or the alloy compound. The used electrolyte is the lithium salt containing solution at the aprotic solvent, for example, LiPF_6 , LiBF_4 , LiClO_4 , LiAsF_6 , LiCF_3SO_3 , $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ In other words, $\text{LiC}(\text{CF}_3\text{SO}_2)_3$ And their mixture.

An plurality of additives used for the lithium ion battery is described in the literature. For example, in EP0759641 and US Patent No.5776627, the anisole in which the organic aromatic compound like the biphenyl substituted thio pen and furan is substituted for in EP0746050 and EP0851524, and mesitylene and xylene derivative are described to be added to electrolyte in order to multiply the stability of battery in case of overcharging. US Patent No.5753389 uses the organic carbonate for the same purpose as additive. The organic boroxine (organic boroxines) is added in EP0856901 in order to improve the cyclic stability. But this all additives have the important defect. Here, the organic material used in the specification referring generally has the low flash point as follows and the low exploding threshold :

Additive.	Exploding threshold (%)	Flash point (°C)
Thiophene.	1.5-1.2	-9
Anisole.	0.34-6.3	43
Mesitylene.	1-6	54
Furan.	2.3-14.3	-35

It is preferable that the electrolyte liquid used according to this situation of this technology the solvent mixture including two or more components. This mixture comprises the strong polar component more than one having the dissociation effect which therefore strong their polarity about the salt. The ethylene carbonate or the propylene carbonate can be in come as the example of this polar component. Because the solvent of the strong polarity has the generally high viscosity, the solvent of the low viscosity is generally added as "diluent" to electrolyte. Typically, 1,2- dimethoxyethane, and diluent such as the dimethyl carbonate and DEC are added to amount of 70% to 30. As to this lower point, the defect of solvent has in the low flash point and the high volatility.

In case of malfunction (the short phenomanon, and overcharging), when the electrolyte solution is electrochemically used as the considerably big extent, the hot formation always occurs and risk toward the combustion of electrolyte increases.

In order that stability is multiplied, cathode and anode space can separate with the microporous separator film. Moreover, by establishing the over-pressure protection apparatus for reacting about the gas emission in overcharging the stability of this battery can multiply.

Fire retardant characteristic phosphorus – and halogen – containing additive are recommended. However, this additive often gives the adverse effect to the battery performance property.

But for such all methods, volatility and flammability "diluent" nevertheless cannot exclude the possibility that it ultimately burns in case of parafunction. It very fierces, the burned lithium reacts to not only the water but also the carbon dioxide.

✧ The Technical Challenges of the Invention

Therefore, an object of the present invention is to provide additive having the conductivity it has the low volatility and the relatively high flash point, and it is physically chemically stable and it is enough mixed with the dissimilar and suitable solvent and excellent.

✧ Structure & Operation of the Invention

The object of the present invention is attained with the compound of the following chemical formula (1) :

X is the H, F, Cl, C in the equation.

nF_{2n+1} , $C_nF_{2n-1}(SO_2)_kN(CR_1R_2R_3)_2R$ Z H, and F or Cl this, and Y H, and F or Cl.

1, R_2 And r_3 H and/or alkyl, and fluoroalkyl or cycloalkyl. $M \geq 0$ and in case X is H, m is not 0. $N \geq 1$.

K 0 in case m is 0. It is 1 in case m is ≥ 1 .

In the compound of the chemical formula (1) is the electrochemical cell like the supercapacitor (supercapacitors) and the first or the second lithium battery, can be used as especially, solvent.

It knew having the flammability in which the compound of the chemical formula (1) lowered. Therefore, the risk of the combustion can be reduced in the malfunction.

Surprisingly, moreover, it knew having the electrochemical stability with high compound of the chemical formula (1). The compound of the chemical formula (1), solvent (example, EC, and DMC) and the typical conducting salt (example of commercial, and LiPF₆) The oxidative decomposition of the electrolyte, including area, at a time, the Li / Li⁺ It experimentally knew treating and arriving in to the electric potential of about 5.5V.

The compound of the chemical formula (1) was known to be mixed with the solvent of commercial. Therefore, as to the phase separation of the conducting salt, moreover, crystallization is not observed.

The compound of the chemical formula (1) can be preferably used moreover, 100% to 1 as amount of 50% to 10 with solvent (the example, EC, DMC, PC and DEC) of commercial in the form of mixture for electrolyte.

The electrolyte which can be used is the LiPF in the aprotic solvent like the EC, DMC, PC, DEC, BC, VC, cyclopentanone, sulfuran, DMS, 3-methyl-1,3-oxazolidine-2-one, γ -butyrolactone, EMC, MPC, BMC, EPC, BEC, DPC, 1,2-diethoxy methane, THF, 2-methyltetrahydrofuran, 1,3-dioxolan, methyl acetate, ethyl acetate and their mixture. 6, LiBF₄, LiClO₄, LiAsF₆, LiCF₃SO₃, LiN(CF₃SO₂)₂In other words, LiC(CF₃SO₂)₃And the solution of their mixture.

Electrolyte comprises the organic isocyanate (DE 199 44 603) moreover, the content of the water is reduced. Similarly, electrolyte comprises the organic alkali metal salt (DE 199 10 968) as additive. The suitable alkali metal salt the alkali metal borate of the following chemical formula (2) :

R the m + p at this time 4 m and p in the equation 0, 1, 2, 3 or 4.

1And r₂ is similar or it is different. And the aromatic hydroxy acid selected from the group consisting of the heterocycle aromatic ring selected from the group, and each case, independently, in other words, together, the coincidence ring by A or Hal to the aromatic hydroxycarboxylic acid and the aromatic hydroxysulfonic acid consisting of the aromatic ring selected from the group, and each case, independently, in other words, together, the coincidence ring by A or Hal to the pyridyl composed according to need with the single bond or the double bond of each case, independently, in other words, together, the aromatic family or the aliphatic carboxylic acid it each other directly unites, the dicarboxylic acid or the sulfonic acid radical, and each case, independently, in other words, together, together, the coincidence ring by A or Hal to the phenyl. Hal F, and Cl or Br. Each case, independently, in other words, together, together, the coincidence ring by A or Hal to the phenyl, is tetrasubstituted or non-substituted naphthyl, and anthracenyl and phenanthrenyl. Each case, independently, in other words, together, the coincidence ring by A or Hal to the pyridyl is tri-substituted or non-substituted, and pyrazyl and dipyridyl. Each case, independently, in other words, together, the coincidence ring by A or Hal to the aromatic hydroxycarboxylic acid and the aromatic hydroxysulfonic acid is tetrasubstituted or non-substituted.

The alkyl having the carbon number of 6 to 1 which A can be tri-halogenated to score monohalogenation.

Similarly, the alkali metal alkoxide (DE 9910968) of the following chemical formula (3) suitables :

In the equation, R the aromatic hydroxy acid selected from the group consisting of the heterocycle aromatic ring selected from the group, and the coincidence ring by A or Hal to the aromatic hydroxycarboxylic acid and the aromatic hydroxysulfonic acid consisting of the aromatic ring selected from the group, and the coincidence ring by A or Hal to the pyridyl consisting of the aromatic family or the aliphatic carboxylic acid, the carboxylic acid or the sulfonic acid radical, and the coincidence ring by A or Hal to the phenyl. Hal F, and Cl or Br. The coincidence ring by A or Hal to the phenyl, is tetrasubstituted or non-substituted naphthyl, and anthracenyl and phenanthrenyl. The coincidence ring by A or Hal to the pyridyl is tri-substituted or non-substituted, and pyrazyl and dipyridyl. The coincidence ring by A or Hal to the aromatic hydroxycarboxylic acid and the aromatic hydroxysulfonic acid is tetrasubstituted or non-substituted.

The alkyl having the carbon number of 6 to 1 which A can be tri-halogenated to score monohalogenation.

Similarly, electrolyte comprises the compound (DE 9941566) of the following chemical formula (4) :

A is the N, P, P (O), O, S, S (O), SO Kt in the equation the N, P, as, sb, S or Se.

2R the , As, the As (O), and Sb or the Sb (O).

1, R2And rThe respective H, the halogen, and substitution and / unsubstituted alkyl C 3 is similar. nH_{2n+1} The substitution of 18 to the , carbon number 1 and/or the nonsubstitution alkenyl, and substitution having the substitution had and/or the non-substituted alkynyl, and one or more triple bonds and/or the non-substitution cycloalkyl C one or more double bonds of the carbon number 1 to 18. mH_{2m-1} A is R in the various location the , mono- or the Polizi ring and/or the nonsubstitution phenyl, and substitution and/or the nonsubstitution heteroaryl.

1, R2And r3Groups which can be included, and Kt can be included in cyclic or the heterocyclic ring, and are combined in Kt may be identical or different. And in 0 persons the case l 1, x, 1 or 2, x 0 or 1 as to n, 18, to 1 m 7, to 3 k is 6, to 0 or 1 l x is 1

Y 4 to 1.

The method for manufacturing the compound characterizes to react the alkali metal salt of the following chemical formula (5) among the salt and polar solvent of the following chemical formula (6).

D in the equation.

Kt, A, and R it is selected from the group consisting of the alkali metal.

1, R2, R3- the , k, l, and x and y defined above statement.

E is F-, Cl-, Br-, I-, BF4-, ClO4-, AsF6-, SbF6-In other words, PF6-It is.

a)The phenol substituted for 3-, 4-, 5-, it is substituted for 6- is mixed in the chlorosulfonic acid and the suitable solvent. The intermediate obtained from the b) a) is reacted with the chloro trimethylsilane. It assorts and processes distillation after filtering the product. The lithium ignition salt of the following chemical formula (7) reacting the intermediate obtained from the c) b) among the suitable solvent with the lithium tetra

methoxyborate (1-) and is manufactured the objective product with the from this favorable method (DE 199 32 317) moreover can exist in electrolyte:

R in the equation.

1And rThe alkyl (C it each other directly can unite according to need with the single bond or the double bond 2 is similar or it is different.

1Inland C.6Alkoxy group (C.1Inland C.6Each case, independently, in other words, together, the alkyl (C the aromatic ring selected with the halogen (F, Cl, Br) from the coincidence ring to the group which is hexa-transposed or composed of the phenyl, naphthyl, and anthracenyl and phenanthrenyl. The phenyl can be non-substituted.

1Inland C.6Alkoxy group (C.1Inland C.6Each case, independently, in other words, together, the alkyl (C the aromatic heterocycle loop selected with the halogen (F, Cl, Br) from the coincidence ring to the group which is tetrasubstituted or composed of the pyridyl, and pyrazyl and pyrimidyl. The pyridyl can be non-substituted.

1Inland C.6Alkoxy group (C.1Inland C.6R the aromatic ring selected with the halogen (F, Cl, Br) from the coincidence ring to the group which is tetrasubstituted. The hydroxybenzenecarboxyl can be non-substituted.

3Inland R.6 is respectively independently in other words each other directly combined to the pair according to need with the single bond or the double bond. And below meaning is had :

1. Alkyl (C.1Inland C.6Alkyloxy (C.1Inland C.6Halogen (F, Cl, Br)

2. Alkyl (C.1Inland C.6Alkoxy group (C.1Inland C.6The coincidence ring by the halogen (F, Cl, Br) to the phenyl, which is hexa-transposed or non-substituted naphthyl, and anthracenyl, phenanthrenyl and alkyl (C.1Inland C.6Alkoxy group (C.1Inland C.6The aromatic ring selected with the halogen (F, Cl, Br) from the coincidence ring to the group which is tetrasubstituted or composed of the pyridyl, and pyrazyl and pyrimidyl. The pyridyl can be non-substituted.

The electrolyte including the lithium or the tetraalkyl - ammonium imide the boron coping with or humanity and justice Lewis acid - solvent addition product, and the ignition salt (DE 199 51 804) of the following chemical formula (8) reacting with the meta nid or tri-plate and is manufactured can be used moreover.

M x, and y in the equation 1, 2, 3, 4, 5 or 6.

E is BR x+ the metallic ion.

1R2R3, AlR1R2R3, PR1R2R3R4R5, AsR1R2R3R4R5And VR1R2R3R4R5R the Lewis acid selected from the composed group.

1Inland R.Alkyl or the alkoxy radical (C which 5 is similar or different, and it each other directly unites according to need with the single bond or the double bond, and can be independently in other words together partly in other words completely substituted for in each case with the halogen (F, Cl, Br): F, Cl, and Br.1Inland C.8Alkyl (C.1Inland C.8The aromatic ring (according to need, it unites through the oxygen): selected from the group consisting of F, cl, the coincidence ring by Br to the phenyl, naphthyl, and anthracenyl and phenanthrenyl or the alkyl (C. The coincidence ring by Br to the phenyl is hexa-transposed or non-substituted.1Inland C.8The

aromatic heterocycle loop (according to need, it unites through the oxygen) selected from the group consisting of F, Cl, the coincidence ring by Br to the pyridyl, and pyrazyl and pyrimidyl. Z performs OR. The coincidence ring by Br to the pyridyl is tetrasubstituted or non-substituted.

6, NR₆R₇, CR₆R₇R₈, OSO₂R₆, N(SO₂R₆)(SO₂R₇), C(SO₂R₆)(SO₂R₇)(SO₂R₈OCOR₆). This, R.

6Inland R. Each case, independently, in other words, together, the hydrogen or R it each other directly unites according to need with the single bond or the double bond 8 is similar or it is different. 1Inland R. 5It the same like the bar defined.

The borate salt (DE 199 59 722) of the following chemical formula (9) moreover can exist :

R_x, and y 1, 2, 3, 4, 5 or 6 M in the equation the metallic ion or the tetraalkylammonium ion.

1Inland R. Alkoxy or the different carboxyl radical (C which is similar or which 4 directly each other can unite with the single bond or the double bond. 1Inland C. 8.

The borate salt is manufactured by reacting 1:1 mixture of lithiumtetraalkoxyborate or the lithium alkoxide and step leak ester among the aprotic solvent to the ratio of the suitable hydroxyl or the carboxyl compound and 2:1 or 4:1.

New compounds can be moreover used for the electrolyte including the lithium fluoroalkyl phosphate (DE 100 089 55) of the following chemical formula (10) :

$1 \leq x \leq 5$, $3 \leq y \leq 8$, $0 \leq z \leq 2y + 1$, ligand (C in the equation.

yF₂y+1-zHzIt is similar or it is different. The compound of the structural formula at this time excludes :

Li+[PFa(CHbFc(CF₃)_d)_e]-

(Here, the sum total of a + e is 6, the ligand (CH b and c are at this time at the same time not 0 a the fixed number of 5 to 2, and b 0 or 1, and c 0 or 1, and d 2, and e the fixed number of 4 to 1 bFc(CF₃)_dIt is each other similar or it is different.

In the hydrogen fluoride one or more compounds of the following chemical formula (11), it with the electrolysis fluorination. It extracts the mixture of the fluorination product which becomes. It makes with the phase separation and/or distillation fractionation. It reacts the fluorination alkylphosphorane of result under the member of the moisture in the aprotic solvent or the solvent mixture with the lithium fluoride and it separates the salt of result with the normal method with refinement but the feature has.

OP(C_nH_{2n+1})₃(IV)

ClmP(C_nH_{2n+1})_{3-m}(V)

$\text{FmP}(\text{CnH}_{2n+1})_3\text{-m(VI), Cl}$

$\text{oP}(\text{CnH}_{2n+1})_5\text{-o(VII) or}$

$\text{FoP}(\text{CnH}_{2n+1})_5\text{-o(VIII)}$

In the equation.

$0 \cdot m \cdot 2, 3 \cdot n \cdot 8$ and $0 \cdot O \cdot 4$.

New compounds moreover can use in the electrolyte including the salt of the following chemical formula (12) (DE 100 16 801) :

$0 \cdot a+b+c+d \leq 5$, $a+b+c+d+e$ is 6, R in the equation.

1Inland R.R it possibles 4 respectively each other independently alkyl, and aryl or heteroarylradical.1Inland R.4The middle two or greater is each other directly combined with the single bond or the double bond.

This compound is manufactured by reacting the phosphorus (V) compound of the following chemical formula (13) under the presence of the organic solvent with the lithium fluoride :

$0 \cdot a+b+c+d \leq 5$, $a+b+c+d+e$ is 5, R in the equation.

1Inland R.4 is the same as defined in above statement.

New compounds can be used for the electrochemical cell electrolyte including the anode material (DE 100 16 024) consisting of the coated metal core which is selected from the group consisting of the sb, bi, cd, in, pb, ga and tin or this alloy. C the b) the suspension the method for manufacturing the anode material manufactures from the suspension of the a) metal core or the alloy core or the urotropine dozing.5-C12There can be the d) metal hydroxide or the feature oxyhydroxide is switched with the thermal process of system into the oxide coping with the c) this emulsion is precipitated on the metal core or the alloy core it emulsifies as – hydrocarbon.

The new compound suspends particle among the lithium–intercalation compound or the organic solvent of commercial. Solution and the particle which is coated after mixing with the hydrolysis solution of the hydrolysis metal compound are filtered. It can be used for the electrochemical cell electrolyte which includes the other cathode material consisting of the lithium mixed oxide particles coated to one or more metal oxides (DE 199 22 522) by incinerating according to drying and need. This is moreover coated to one or more polymers (DE 199 46 066). It can be composed of the lithium mixed oxide particles obtained with the method for filtering the particle suspending particle in solvent and is coated and dry and incinerating according to need. Similarly, the new compound can be used as the alkali metal compound and metal oxide (DE 100 14 884) for the system including the cathode consisting of the single or the multi-coated lithium mixed oxide particles. The method for manufacturing this material filters the particle adding the metal oxide suspending particle in the organic solvent and adds the suspended alkali metal salt compound and dissolves in the organic solvent among the organic solvent and mixes the suspension with the hydrolysis solution and is coated. It incinerates with drying but the feature has.

Therefore, the present invention is to provide the electrolyte including the compound of the chemical formula (1).

Moreover, the present invention is to provide the electrochemical cell, consisting of the reported cathode, and anode and separator especially, the first, the second lithium battery and supercapacitor. The reported cathode obligatorily copes with.

It mixes with the conducting salt of commercial and the compound of the chemical formula (1) offers the excellent conductivity.

The general working example of the present invention is hereinafter little more particularly explained.

The manufacture of the compound of the chemical formula (1).

The apparatus comprised of mixer and cooling means was charged to dimethylamine among the suitable solvent. The suitable solvent the organic solvent, for example, diethylether or the chloroform.

Partly, while fluorination or the perfluorinated alkylsulfonyl fluoride being ***ed at a temperature of -30°C to 0°C and cooling, it added. Next, and the reaction solution were warmed to the room temperature to 40°C temperature. Solvent was by distillation removed.

But it possibles to react the halo sulfonamide with the fluoridising reagent of commercial, for example, the antimony trifluoride, and the arsenic trifluoride or the potassium fluoride.

In apparatus comprised of condenser the halo sulfonamide among the suitable solvent like the benzene and mixer, it refluxed for 4 hours (preferably, 2 hours) to 1 while ***ing with the fluoridising reagent. The reaction solution was cooled to the room temperature and it was filtered. Solvent was by distillation removed and residue was distilled under the pressure reduction. According to need product, it made re-distill under the atmospheric pressure.

Flammability.

The flammability of the compound of the chemical formula (1) was examined. It is the thing to burn the compound manufactured according to the process of being above mentioned with the open air flame. But this attempt was unable to succeed.

Electrochemical stability.

LiPF the compound of the chemical formula (1) in the aprotic solvent like the EC, DMC, PC, DEC, BC, VC, cyclo pentanone, sulfuran, DMS, 3- methyl -1,3- oxazolidine -2 -one, γ - butyrolactone, EMC, MPC, BMC, EPC, BEC, DPC, 1,2- diethoxy methane, THF, 2- methyltetrahydrofuran, 1,3- dioxolan, methyl acetate, ethyl acetate and their mixture.6, LiBF_4 , LiClO_4 , LiAsF_6 , LiCF_3SO_3 , $\text{Li}(\text{CF}_3\text{SO}_2)_2$ In other words, $\text{Li}(\text{CF}_3\text{SO}_2)_3$ And it added in the electrolyte consisting of the conducting salt of the commercial like their mixture. Amount of the compound of the chemical formula (1) among the solvent mixture 100% to 1.

In measurement battery containing the stainless steel 5 cyclic voltammogram to 3 as to each case, the platinum or the gold working electrode, and the lithium counter-electrode and lithium reference electrode, it and then recorded. The Li / Li it naturally starts as to this purpose from the electric potential (rest potential).+It naturally again returned to the electric potential after multiplying to voltage more than the decomposition potential of each of the additive which it treated and which coped with to the rate of $1\text{mV} / \text{s}$ to $100\text{mV} / \text{s}$.

The oxidative degradation of electrolyte from this result, area, at a time, the Li / Li.+It could know treating and arriving in to the electric potential of about 5.0V. Therefore, these suitables for the electrochemical cell to use.

The conductivity of scrambling with the standard solvent and result.

The LiPF in the aprotic solvent like the EC, DMC, PC, DEC, BC, VC, cyclo pentanone, sulfuran, DMS, 3- methyl -1,3- oxazolidine -2 -one, γ - butyrolactone, EMC, MPC, BMC, EPC, BEC, DPC, 1,2- diethoxy methane, THF, 2- methyltetrahydrofuran, 1,3- dioxolan, methyl acetate, ethyl acetate and their mixture.6, LiBF₄, LiClO₄, LiAsF₆, LiCF₃SO₃, LiN(CF₃SO₂)₂In other words, LiC(CF₃SO₂)₃And while gradually multiplying the amount of the compound of the chemical formula (1), it added in the standards electrolyte consisting of their mixture.

As to the phase separation of the conducting salt, moreover, crystallization was not observed. The compound of the chemical formula (1) was mixed to the random quantity with the standards electrolyte.

In the conductivity experiment is the various temperature, it enforced by using the standards electrolyte.

The following embodiment is not thus limited to the thing for little more particularly explaining the invention.

Working example.

Working example 1.

N,N- dimethyltrifluoromethylsulfonamide.

In 3- neck flask equipped with the Cold Trap, and the means for introducing mixer and gas reagent, the reaction was enforced. The Cold Trap was kept with the temperature of -78°C. The diethylether of 250cm³ was introduced to flask. It cooled to the ice water. The gas property dimethylamine of 138 g (3.07 mole) which was obtained from the reaction of the saturated water oxidation sodium solution of the dimethylamine hydrochloride of 260 g (3.19 mole) and 153 g (3.83 mole) and was built on the potassium hydroxide was condensed in flask. It added 2 for hour while ***ing the gas property trifluoromethyl sulfonyl fluoride of 202 g (1.33 mole). After the addition of all reagents was accomplished, the reaction container was warmed to 2 for hour 40°C. It extracted in diethylether after diluting the reaction mixture to the water of 0.5ℓ. MgSO the extracts is washed to the water.4It desiccated in phase. Diethylether was by distillation removed and residue was distilled under the atmospheric pressure.

The N,N- dimethyltrifluoromethylsulfonamide of 230.4g was obtained.

Yield: 98%.

CF₃SO₂N(CH₃)₂: 152°C to the boiling point 151.

¹⁹F-NMR: -75.1 seq(CF₃SO₂)

¹H-NMR: 3.05q(2CH₃)

JH,F= 1.2 Hz

Working example 2.

N,N- dimethyl nonafluorobutyl sulfonamide.

In 3- neck flask equipped with the Cold Trap, and mixer and dropping funnel, the reaction was enforced. The Cold Trap was kept with the temperature of -78°C. It added in -30°C in the liquid dimethylamine of 43 g (0.95 mole) while ***ing the perfluorobutyle sulfonyl fluoride of 100 g (0.331 mole). After addition was accomplished, the reaction mixture was warmed to the room temperature and it ***ed 3 for hour. The water of 0.1ℓ was added

and the mixture of result was extracted and then in diethylether. Na the extracts is washed to the water. H_2SO_4 it desiccated in phase. Solvent was by distillation removed.

The N,N- dimethyl nonafluorobutyl sulfonamide (white crystal quality) of 114.5g was obtained.

Yield: 87.5%.

$\text{C}_4\text{F}_9\text{SO}_2\text{N}(\text{CH}_3)_2$: melting point 32°C .

^{19}F -NMR: $-81.5\text{t}(\text{3F, CF}_3)$, $-112.2\text{t}(\text{2F, CF}_2)$, $-121.9\text{m}(\text{2F, CF}_2)$, $-126.4\text{t}(\text{2F, CF}_2)$

$^3\text{J}_{\text{F,F}} = 2.2 \text{ Hz}$

$^4\text{J}_{\text{F,F}} = 9.9 \text{ Hz}$

$^4\text{J}_{\text{F,F}} = 13.9 \text{ Hz}$

^1H -NMR: $3.1\text{s}(\text{2CH}_3)$

Working example 3.

Bis (N,N- dimethylaminosulfonyl) difluoromethane.

In 3- neck flask equipped with the Cold Trap, and mixer and dropping funnel, the reaction was enforced. The Cold Trap was kept with the temperature of -78°C . It added in -30°C in the liquid dimethylamine of 101 g (2.084 mole) among the chloroform of 100cm^3 while adding the di (fluorosulfonyl) difluoromethane of 99 g (0.458 mole). After addition was accomplished, the reaction mixture was warmed to the room temperature and it stirred 3 for hour. Solvent was by distillation removed. The water of 0.3l was added in residue. The mixture of result was extracted and then in diethylether. Na the extracts is washed to the water. H_2SO_4 it desiccated in phase. The solvent of about 80% became with distillation elimination.

The bis (N,N- dimethylaminosulfonyl) difluoromethane (white crystal quality) of 91.2g was obtained.

Yield: 74.8%.

$(\text{CH}_3)_2\text{NSO}_2\text{CF}_2\text{SO}_2\text{N}(\text{CH}_3)_2$: 72°C to the melting point 71°C .

^{19}F -NMR: $-100.4\text{m}(\text{2F, CF}_2)$

^1H -NMR: $3.06\text{t}(\text{4CH}_3)$

$^5\text{J}_{\text{H,F}} = 1.0 \text{ Hz}$

Working example 4.

N,N- dimethylaminosulfonyl fluoride.

In 2- neck flask equipped with mixer and cooling means, the reaction was enforced. The N,N- dimethylaminosulfonyl chloride of 80 g (0.557 mole) among the anhydrous benzene of 100cm^3 was added in the antimony trifluoride of 66 g (0.369 mole). It added while adding 5 fluorinate antimon of 5cm^3 . The reaction mixture was refluxed 2 for hour. Solution was cooled to the room temperature and it filtered. After the

benzene was by distillation removed, residue was distilled under the pressure reduction. It redistilled under the atmospheric pressure and the pure N,N- dimethylaminosulfonyl fluoride of 53.8g was obtained (R. Heap, J. Chem. Soc. 1948, 1313)

Yield: 76%.

FSO₂N(CH₃)₂: 150°C to the boiling point 149.

¹⁹F-NMR: 33.0 seq

4JH,F= 2.0 Hz

Working example 5.

The flammability of the N,N- dimethyltrifluoromethylsulfonamide.

Burning the N,N- dimethyltrifluoromethylsulfonamide of 100mℓ of airborne with the open air flame was. This attempt was unable to succeed.

Working example 6.

The electrochemical stability of the N,N- dimethyltrifluoromethylsulfonamide.

In measurement battery containing the platinum electrode, and the lithium counter-electrode and lithium reference electrode, 5 cyclic voltammogram was recorded and then. The Li / Li it naturally starts for this purpose from the electric potential.+After treating and multiplying to the rate of 10mV / s to 6mV / s, it naturally again returned to the electric potential.

Profile was obtained. The LiPF of the Gram formula weight / ℓ among the EC / DMC / N,N- dimethyltrifluoromethylsulfonamide (47.5/47.5/5).6The oxidative degradation of the composed electrolyte, shift, at a time, the Li / Li.+It treated and it arrived in to the electric potential of about 5.5V. Therefore, electrolyte suitable for the lithium ion battery including the transition metal cathode to use.

Working example 7.

The conductivity of miscibility with the standard solvent and result.

It is reported the standards (1: the LiPF of the Gram formula weight / ℓ among the EC / DMC of 1.6ℓ added while gradually multiplying amount of the N,N- dimethyltrifluoromethylsulfonamide. The fluorination solvent was mixed to the random quantity with the standards electrolyte. As to the phase separation of the conducting salt, moreover, crystallization was not observed.

Conductivity data :

Electrolyte: the LiPF of the Gram formula weight / ℓ among the EC / DMC / N,N- dimethyltrifluoromethylsulfonamide (47.5/47.5/5).6

Temperature (°C)	20	-20	-30
Conductivity (mS/cm)	8.6	2.9	2.1

■ Effects of the Invention

Provided is the electrochemical cell having the low volatility and the relatively high flash point by using the electrolyte solution including the compound of the new chemical formula (1) of the present invention, and is physically chemically stable, and miscibility with the other suitable solvent excellents, the conductivity excellents.

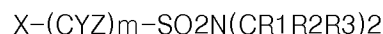


Scope of Claims

Claim 1 :

The compound of the following chemical formula (1) :

Chemical formula 1.



X is the H, F, Cl, C in the equation.

nF_{2n+1} , $C_nF_{2n-1}(SO_2)_kN(CR_1R_2R_3)_2$ Z H, and F or Cl this, and Y H, and F or Cl.

1, R_2 And r_3 H and/or alkyl, and fluoroalkyl or cycloalkyl. M 9 to 0 and in case X is H, m is not 0. N 9 to 1.

K 0 in case m is 0. It is 1 in case m is 9 to 1.

Claim 2 :

Partly, the method for manufacturing the compound according to the first claim wherein fluorination or the perfluorinated alkylsulfonyl fluoride is reacted among the organic solvent with dimethylamine.

Claim 3 :

The method for manufacturing the compound reacting the halo sulfonamide among the organic solvent with the fluoridising reagent of commercial according to the first claim.

Claim 4 :

The method for being selected from the group of claim 2 or 3, wherein it is composed of the organic solvent is diethylether, and the benzene and chloroform.

Claim 5 :

The use of the compound according to the first claim used in the electrochemical cell electrolyte as the low flammability solvent.

Claim 6 :

The use in which the compound is used as to claim 5 with the solvent of the other commercial.

Claim 7 :

The electrolyte including one or more compounds according to the first claim.

Claim 8 :

The electrochemical cell consisting of cathode, anode, and the electrolyte according to separator and claim 7.

Claim 9 :

The lithium battery according to claim 8 and supercapacitor.



Drawings

Fig. 1

